Notebook Series -- Notebooks by Experimenters Other Than Edison
Chemical Experiments -- Miscellaneous Books
Notebook N-14-12-16.2

This notebook was used during December 1914-November 1915, probably by Peter C. Christensen, for notes on chemical experiments. The entries describe various batches of chemicals, including paranitroacetanilide, nitrobenzene, aniline, acetanilide, chloronitrobenzol, and para-phenylenediamine ("para"). Some of the entries include notations by Edison. Inserted into the book is a large drawing by Edison of the production flow for "mother liquor," along with several other items bearing notations by Edison. The front cover is marked "1." The spine is labeled "Para - 1." The book contains 202 numbered pages, many of which are blank.

63224

Arme Co.,

MFG. STATIONERS,
96 JOHN ST.
AND
19 PLATT ST.
NEW YORK.

~~D.R.~~ D. 42011 - 42814

100 L H_2O
100 Kg Iron
40 L HCl
400 K Water
300 Iron

Recived Raw Material Feb 16 - 1914

500	Lbs of	Acetanilid
1000	" "	Soda Ash
4650	" "	Iron borings
6	Carboy.	Nitric Acid
2	"	Acetic "
12	"	Muriatic Acid
1500	Lbs	Sulphuric Acid
310	Lbs	Sodium Sulphite

1

Nitrating Acid Batch #1 - 12-17-1
1 Part of HNO_3 + 2 parts H_2SO_4
369 lbs of Nitric Acid
425 " " Sulphuric Acid
left to cool over night

2

Batch #1 - started 12-18-14
65 liters of H_2SO_4 sp. G. 1830 at 10°C
we add 14 liters more making 75 liters
67.5 lbs of Acetanilid was sprinkled into thick H_2SO_4 while continuously stirring and not allowing temperature to rise more than 30°C - then cool down with water-jacket, till 15°C.
Then add very gradually 140 lbs of Nitrating acid not allowing temperature to rise higher than 30°C at the very most. Temperature kept down by steam & water. After all acid has been put in solution is still being stirred 2 hours longer, after that it is left stand over night.

Now the nitrated solution is put into water and ice when it is precipitated here also is the temperature kept down to not more than 80°C the yellow precipitate is transferred to a filter and washed free from Acid. This Paranitroacetanilid is put into an iron tank to be reduced by Iron 75 lbs & 7½ lbs of Acetic Acid that boiled and stirred very well - add 5 lbs of Acetic acid over.

Batch #1. continued

stirred and boiled well for several hours, add 5 lbs of Acetic. Stir and boil and let stew for night — next day boil up again and neutralize with 8 lbs of Soda ash. stir & let all day and let stay over night — next day boil up again adding 15 lbs Acetic Acid when boiling add 20 lbs of Lime & 15 lbs of Acetic boil all day and kept hot, let over night — adding 12 more lbs of Acetic Acid boil all day and kept hot over night again — following day it was neutralized with 24 lbs of lime, boiled well for 2 hours and filtered boiling while being filtered into wooden tank, which it was evaporated down, and put into crok, where we got of first crop 28 lbs of Acetyl para Phenylenediamin, these was centrifuged free from water, — and put into kettle and boiled them in 37 lbs of 20% Proof 1 gal of water for several hours, then tested and it showed good paraphenylenediamine it was now desirable for economy of labor to put the second crop and all Mother

Batch #1 continued

liquor + 37 lbs of 20% Proof to gether with the first lot and boil them 2 more times and while hot transfer it all into crok —

#1 Crystalization from Crok, = 28 lbs wet

#2 " " first Mother liquor Partie Crystals

outside in in cold — the last Mother liquor was put in with Batch #3 —

Dry Crystals (same concentration) 25 lbs

" " second crystalization 10 "

35

Total

Batch # 2

65 Lbs H_2SO_4 same as used in # 1 Batch
67.5 Lbs of Acetonitril
Stirred and kept pretty cold up to
about 70°C - Acetonitril should dissolve
very well in this way a little warmer
better,
135 Lbs of Nitrating acid from Batch
#1 was added it took 5 hrs to get
it all in

Precipitate & Wash -
Reduction

75 Lbs of Iron } added in
30 " 1/4 Acetic Acid } portions
15 Lbs of Soda ash
boiled again for half hr.
Then filtered and gave 150 Lbs of Soda
Ash and the Nitrobenzene from Batch
#1 - evaporated and put into crocks

Batch # 3

We made the Nitrating Acid separate
taking 46.5 Lbs of Nitric Acid 67.5% (42° Be)
" 91.0 " " Sulphuric " (66° Be)
cool down overnight

67.5 Lbs of Acetanilid
270 Lbs = 86 Lbs H_2SO_4 66° Be

This Nitration was not allow to go
higher than 65° F.

The precipitating of the above was
also kept low (80° F) - after washed
free from Acid, it was reduced as follows

25 Lbs Iron 10 Lbs Acetic acid some water
stir & boiled well
25 Lbs Iron & more of the ppt
stir & boil well
25 Lbs Iron & 5 Lbs Acetic Acid and
more ppt - boil & stir
25 Lbs Iron & 5 Lbs of Acetic Acid
and the last of the ppt the operation
lasted about 14 hr, the stuff was
stirr and boiled from 10 Am till 600 PM

Batch #3 con-
and kept hot over night,
following morning we neutralized the
Iron with 8 Lbs of Soda ash ppt -
[illegible] 200 Lbs
200 Lbs Soda, evaporated and put into crocks
for crystalizing

Dye Crystal 38 Lbs

270

Batch # 4 (First Day)

66 Liters of H_2SO_4 + 67.5 Lbs Acetanilid started at 8.15 a.m. finished 9.30 a.m. (1 hr 15 min) tept 85 °F

Nitration started at 2.00 P.M. and finished at 7.15 P.M. (5 hr 15 min)

Second Day

Precipitation started 8.30 a.m. and finished 9.45 (1 hr. 15 min) 2 men for this operation — left stay till 12 o'clock then put on filters which took 1 hr. washing all afternoon

third day

starting washing again all day

fourth day

started washing again 3 times and afternoon was used for Reduction which was finished after 5 hrs, and left it stand over night & Sunday then

fifth day

Iron precipitated with 7 Lbs of Na_2CO_3 boiled 1/2 hr. filtered saponified with 200 lb of NaOH 20% ——— . First day crystals taken off - centrifuge - put in oven over night then cooled

JAN 1 - 1915

Batch # 5

Operation & Remark on Para	Time
Starting 7.30 a.m. - weigh off H_2SO_4 and putting into pot weighing off Acetanilid and putting into pot P.S. it takes 1 hr. to put the acetanilid in highest temperature 85 °F.m.	2 1/2 h.
Nitration started at 1.20 P.M. finished 5.45 using N.A Batch #1 - 120 Lbs (75 °F)	4 hr.
Precipitating next day settle till 11 a.m. - 1 Transferred over to filter box washed 2 days	First Day 2 hrs 2 days
Reduction started 3.00 P.M. finished 6 P.M. 20 Lb Acetic Acid 25 Lbs of Iron - left stand over night	1 day
Precipitating Iron with 7 Lbs of Na_2CO_3 boil 1 hr. then filter & wash - leave stay in tank over night	1 day

#1	Dissolving acetanilid	5 hr	1 Day
" 2	Nitration	5 "	
" 3	Precipitating	5 "	1 Day
" 4	Washing	5 "	
" 5	Reducing	10 "	1 Day
" 6	Precipitating Iron-filtering and washing	5 "	1 Day
" 7	saponifying & evaporation	5 "	
" 8	Crystals taken out centrifuged & washed product been & Drying		?

Batch #6 started 1-4-15

66 liters of H_2SO_4 66° Be } morning
67.5 lbs " Acetamilid
120 lbs of " Nitrating Acid } afternoon

Precipitating — morning

1-11-15

Monday 7.00 A.M. - Batch - # 9 - Started starting with washing of 66 liters of Acid and 67. Lbs of Acetanilid - 1 and also the Titrating Acid - From dissolving of Acetanilid was started after that the Carbonate was added to # 7 Batch & boiled for some time the lump of Acetanilid was crushed and pot was cooled down to get ready for Titration at 1.00 o'clock - From the drawing off of # 7 commenced and the washing of residue and the washing of Batch # 8 was continued all day when a chance accurd

stoped at 6.00 P.M.

1-12-15

Tuesday 7.00 A.M. Batch # 9 starting with precipitating Batch 9 - - two from 70 do this job more washing of Batch # 8 - at 1 P.M. we started to Reduce # 8 - two men on this job some work is run on Centrifugal Batch 5-6 - and before 6.00 o'clock we end the precipitated Batch on Filter Press

stoped at 7 o'clock

1-13-15

Wednesday - Batch #9

Started to make Batch #10 - first having trouble with water in Kettle from Rain over Night - Batch #8 was not reduced yet so I stirred it up afterwards having steam on all day - I also washed #9 all day say 5 times, (Fred) centrifuged Batch #1, 5-6 -,

A long time is consumed by centrifuge

stoped work at 9.00 P.M.

1-14-15

Thursday - Batch #9

started at 7.00 A.M. to Precipitate #10 Batch

Batch #8 was not reduced yet so I had to get a Man for stirring it up and 5 Lbs of Iron again - after it was fully reduced we forgot to put in Soda till half had been removed to tank so we had to return all into kettle again, - and after this we put in the Na_2CO_3 boil 1/2 hour then transferd over into tank, - washed the sediment 3 times - it was then 6.00 P.M. - We washe Batch #9 2 times in the Morning -

stoped at 6.00 P.M.

1-15-15

Friday - Batch #9

Starting Batch # 11 at 7.00 A.M.
and Reducing # 9, 1 man Stirring all day
Transferring Batch #10 into filtering boxes
and starting to wash. - Caustic Soda
~~was~~ was put into Batch # 8 - and boiled
down - and put into pots.
We must get less water in the Soda
Solution, as it takes too long to
evaporate it down.

Stopped at 7.30 P.M.

1-16-15

Saturday Batch # 9 -

Starting 7.00 A.M.
Precipitating # 11 and washing # 10 Batch that
finished precipitated Iron in Batch #9
filtered it ~~and~~ into Boxes where it stays
over night, Continued Batch # 11 precipitating
stopped 7 P.M.

1-17-15

Sunday Batch #9

Started at 8.00 A.M.
Saponifying #9 - and put it in Pots, and washing out residue and Centrifuge work of #7 Batch
~~Reduce #10 - and transfer #11 Batch~~
stopped at 5.00 P.M.

1-18-15

Monday Batch #9

Started 7.00 A.M. Making #12 Batch
Reducing #10 Batch Transfer #11
washing #11 - centrifuge work of #9 Batch
these are now drying -
stopped at 9.30 P.M.

1-19-15

Tuesday

9-Batch Drying

Started 7.00 P.M. to precipitate #12 Batch
10 Batch was not complete reduced
it was started at 9.a.m. yesterday
and stood all day & night over night
with some steam on,
it took till 1.00 P.M. to day
before it was complete Reduced.
after full steam and continuously
stirring washing of # 11 Batch
was finished washing at ~~4.00~~ 2.00 P.M.

Mother liquor of 1-7-Batches

Boil with more Soda

Regarding Centrifuging Paranitroacetanilid

I took a sampl of our Batch #29 which is 67.5 lbs of Acetanilid nitrated and precipitated in 150 gal of water centrifuged 6 times. 5 times added its own bulk of water,

670 cc. of water was flung out of 13300 cc of same Batch as above after it was all finish washed and settled in filterboxes our Filterboxes holds 32 pails each pail = 12 L × 32 = 384 ÷ 16 L of water added to get stuff out of pail

384
16
2 | 368
184

it means that the paranitro having a space of 368 L by centrifuging removes 184 L of water

or our last wash can be to advantage be increased in bulk to replace the water

(1 gal of washed & settled weighs 4100 grams

Regarding Yield of Paranitroacetanilid

Took 200 cc of Batch #30 - direct from nitrating pot 350 gr is the weight of 200 c.c. when 67.5 lbs Acetanilide is dissolved in 66 lbs of 66° Bé H_2SO_4
This was precipitated in 1300 cc. water kept no higher 25°C.
now test for yield - after centrifuging and absolute drying I get 45.2 grams of Paranitroacetanilid

Acetanilid for Nitration

First Test

67.5 - 6.66 1 - 1.5

1 lb Acetanilid

1 lb 667 g.

67.5 - 90 - 1.2

67.5 / 900 (1.333 lb)
675
2250
225
2250
2025
225

454
1.333
1362
1362
1362
454
605.182 gm

67.5 = 45 - 1

67.5 / 450.0 (.666
405.0
45.00
45.0
45.00

67.5 / 100 (1.037
67.5
25.00
2025
475

454
.666
2724
2724
2724
302.364 gr

454
1.04
1816
454.0
472.16 gr

605 / 485.00 (80.1
484.0
1.010

Made same as our Big Batches
it takes 12 gal, of water to wash 1 lb of Acetanilid when nitrated free from acid
yield 485 gr = 80 %

see page 29

Paranitroaniline

1 gal of Paranitroacetanilid = 410 gram
Mother of liquor which is to be reduced - it contains 50 % of water King says 1 gal contains 1 lb of solid -
(1 lb of solid 450 g of 15 % NaOH
started at 10.30

an attempt was made to stir it by motor but broke down so it was left it without an at. 9.00 o'clock following day we took it of hot plate
tipt. 55 °C (it took 24 hr for operation)

another Batch same as above was started at 3.00 P.M.
and finished at 10.00 A.M. (1 day)

yield of first Batch 375 gr from centrifuge
300 gr Dry = 85 %
" " second Batch 265 gr Dry

Acetanilid for Nitration

con. from page 27

first batch was allowed to stand for night in glass jar (the precipitate was yellow) 1 Lb Acetanilid gives
455 g paranitroacetanilid
350 g paranitroaniline

#2 Batch

second test or 2 Batch was made same as #1 but by mistake 40 grams of nitric acid above the right amount was added, but it was precipitated at once. This batch was not dried but put right through 1 Lb of Acetanilid gave
365 g paranitroaniline

#3 Batch

was nitrated with same proportions as #1 but not allowed to stand overnight ~~with~~ in contact with Iron, it was put 1 hr after finished.
1 Lb Acetanilid
455 g paranitroacetanilid
370 g paranitroaniline

Paranitroaniline in Benzol
to recover P.

4,600 c.c Total liquor
500 cc of liquor shaked with 3200 c.c. Benzol and distilled of left a part of para- and some black residue also some oil. Total weight from 500 c.c. was 1.3 g or 1 lit 2.3 g = 11.96 g in 4.600 c.c. ?

#4 Batch

1 Lb of Acetanilid } dissolve (tept 25°C)
1 L of 66° Be H_2SO_4 } absolute no higher
now reduce tept to zero Centigrade and while nitrating dont allow to rise to more than 15°C -
325 g 65.2 % Nitric acid
500 g " 66° Be H_2SO_4

#5 Batch

same as #4 only using 1½ L of H_2SO_4

#6 Batch

same as #4 only using 500 c.c. H_2SO_4

over

Nitrating Acetanilid

Dup Batch #4
but Centrifuge just once

Batch # 4-5-6 (of Dry) are not Mixed

Nitrating Acetanilid

Using Crud Acetanilid → 90% pure
504g Acetanilid / 1 Liter 66° Be H_2SO_4
505g 66° Be H_2SO_4
325g 65.2% HNO_3

504g 90% = 1 Lb Acetanilid
515g Paranitroacetanilid

This is = 85% yield
provided that Acetanilid is 90%
and that the Nitration is complete

To make Para —
67.5 = 90 = 227 = 302 Paranitroacetanilid + 500 cc water

90 − 10 = 300 = 33.3 of 28% Acetic Acid

$\frac{33.3 \times 28}{70}$ = 13.2 g of 70% " " (Recovered)
Added 50 cc of Water to acid

90 − 75 = 300 = 250g Iron filling 100 cc Water

90 − 30 − 300 = 500 g of 20% Soda gel 180g Wet
evaporated to 1,000 cc.
washed with 200 cc
got Crude 102g Dry

m.p. 138°

Paranitroaniline ($C_6H_4NO_2NH_2$)

(from Crud acetanilid 90%) m.p. 147

227g Paranitroacetanilid + 1.5 L of Water
325g " 18% Naoh

stirred 5 hr. & kept 55°C got 220g Wet
" 161 Dry crude

Mother liquor 2 Litres
Washed with 1 " water

Recrystalized

7500 cc of Water — 200g Wet
6,600 cc of Mother Liquor 155 Dry

Para

Extracted 11 times with 400 cc Benzol
weight of Residue 29g Crude
250 cc 28% Acetic Acid to Neutralize Mother Liquor
300 g dry Sodium acetate Fused 200 gr from this
Daily made glacial Acetic Acid

Batch #	Dissolv Acetamide	Precipitate	Precipitate	Washing Precipitate	Washing Precipitate	Filtering	Filtering	Centrifuge Sacrify	Crystalizing	Centrifuging	Drying	
12	6½ hrs	5½ hrs	2 hrs	24	26	2	2	0	36	3	18	#12 on Reducing coil burstin[illegible] steam in[illegible]
13	5 "	5 "	2 "	8	15	1	2	10	36	3	20	#13 1 man washing & stirring all the work
14	5 "	6 "	2 "	24	12	1	2	6	36	3	20	
15	5 "	5 "	2 "	18	12	1	2					
16	5 "	5 "	2 "	18	4	1	1					
17	5 "	5 "	2 "	15	3 10-a							
18	5	5 "	2 "	15	3 15-a							
19	5 "	5 "	2 "	10	3½ 10-a							
20	5 "	5 "	1½ "	10	2 5							#20 was not reduced after 5 hrs. [illegible]
21	4 "	5 "	1½ "	10	7 5							
22	5 "	5 "	1½ "	10	2 5							
23	5 "	5 "	2 "	10	8 6							
24	5 "	5 "	2 "	10	6 10							
25	5 "	5 "	2 "	10	4 10							
26	5 "	5 "	2 "	10	4 10							
27	5 "	5 "	2 "	10	4 10							
28	5	5	2 "	10	8 7½							
29	5	5	2 "	10	4 10							
30	5	5	2 "	10	4							
31	5	5	2 "	10	4							
32	5	5	2 "	10	4							

Date 1915	Batch #	66° Be 7.50 lbs	Acetanilide	70%-42° Nitrating acid	Ferrous	98% Acetic acid	$NaNO_3$	Nach 20%	Para yield
13-14	1	75 lbs	67.5 lbs	140 lbs	95 lbs	59	32	74 lbs	~~35 lbs~~
" 21 "	2	65 "	67.5	138 "	75 "	30	15	150 "	35 "
" 25	3	66 "	67.5	136.5 "	100	20	5	200	38 "
" 27	4	66 "	67.5	136.5	75	20	7	200	77 "
1-1-15	5	66 "	67.5	120 "	65	20	7	200	72
" 4 "	6	66 "	67.5	120 "	65	22	7	200	(5-6-7)
" 6 "	7	66 "	67.5	120 "	60	20	7	150	
" 8 "	8	66 "	67.5	136.5	65	20	7	200	
" 11 "	9	66 "	67.5	136.5	60	20	7	200	omit 8-9-10 76 lbs
" 13 "	10	66 "	67.5	136.5	60	20	7	200	8-9-10 115
" 15	11	66 "	67.5	136.5	65	20	7	200	
" 18	12	66 "	67.5	106.5	65	20	7	200	(11-12) 67
" 20	13	66 "	67.5	106.5	75	20	7	200	40
" 22	14	66 "	67.5	106.5	75	20	7	200	40
" 25	15	66 "	67.5	106.5	75	20	7	200	39
" 27	16	66 "	67.5	106.5	75	20	7	200	36
" 29	17	66 "	67.5	106.5	75	20	7	200	40
2-5-15	18	66 "	67.5	106.5	75	15	5	200	40
2-10-	19	66 "	67.5	95.0	75	10	2½	200	36
2-24	20	66 "	67.5	95.0	75	5	2	0	43
2-25	21	66 "	67.5	95.0	75	5	2	0	43
2-26	22	66 "	67.5	95.0	75	5	2	200	43
3-1-3	23	66 "	67.5	45+75	75	6	2	200	43
2	24	66	67.5	45+70	75	7.0	2	200	43

Hour Test	Melting Points	Special Numbers	M.P.	Hour Test	Remarks
22 hrs	110-115	Dehydrated	137-138		Reg Dehydrated 12 hrs
16 "	136-138	Crude	134-136		
20 "	130-135	3-A	137-139	16	
12½ "	134-136	5 A		26 "	#4 two parts [illegible]
13 "	134-38	5 B		12½ "	#5-A has not been centrifuged #5 B has been recrystallized
					(2-5-6-7-) in 1 BBL
					(-11-12-13 in) 1 BBL
11½	136-139				(13-14-15-16-) 1 BBL
					17
13 "	134-138				
13 "					(20-21-22-) 130 lbs
14½ "					
14½					(23-24-25) 131 lbs
14 "					(26-27-28-29) 132 lbs
14½ "					
13					
13					total on hand 535 lbs
14½					
13					
14					
13					
13					

Date 1915	Batch #	C_6H_2	Wet - H_2SO_4	Wet - Commercial	Strength of Acid 60% H_2SO_4	Strength of Acid 70% HNO_3	Acetic Acid	Iron turnings	$NaNO_3$	NaOH 20%	Para yield	Test Hrs	M.P.	
3-11	25	66	67.5		70	45	10	75	2	200	43.5	12		
3-16	26	66	67.5		70	45	10	75	2	200	36	12		20 lbs of this sent to New York
3-19	27	66	67.5		70	45	10	75	2	200	40	13		
3-22	28	66	67.5		70	45	7.5	75	2	200	38.5	13		
3-24	29	66	67.5		70	45	10	75	2	200	38.5	13		
3-26	30	66	67.5		70	45	10	75	2	200	39	13½		
" 31	31	66	67.5		70	45	10	75	2	200	42	13½		
" 2	32	66	67.5		70	45	10	75	2	200	41½	12½		
4-5	33	66	67.5		70	45	10	75	2	200	41.5	13½		10 lbs of this sent to New York
7	34	66	67.5		70	48½	10	75	2	200	40	12		
9	35	66	67.5		70	48	10	75	2	200	39	12½		
12	36	66	67.5		70	48	10	75	2	200	42½	14½		
14	37	"	"		"	"	"	"	"	"	38	13		
[illegible]	38	"	"		"	"	"	"	"	150	38	13		
21	39	"	"		"	"	"	"	"	125	39	12		
22	40	"	"		"	"	"	"	"	"	39	13		
26	41	"	"		"	"	"	"	"	"	37	12		
28	42	"	"		"	"	"	"	"	"	42½	14		
30	43	"	"		"	"	"	"	"	"	38½	14		Delivered May 22-15
5-29	44	"	68		"	"	"	"	"	"	31.5	11		using crude Acetanilid
31	45	"	"		"	"	"	"	"	"	40.5	12½		" " " half an hour pur
6-3	46	"	"		"	47½	"	"	"	"	41.5	13½		using crude Acetanilid
6-5	47	"	"		"	"	"	"	"	"	45.5	12½		
6-7	48	"	"		"	"	"	"	"	"	42.5			

Date 1915	Batch #	66% H_2SO_4	Acet anilid	Nitrating Sol 66% H_2SO_4	70% HNO_3	Acetic Acid	Iron filings	$NaClO_3$	NaOH 20%	Para yield	Test	M.P.		Remarks
6-15	49	66	67.5	70	47.5	10	75	2	125	44				
" 16	50	66	67.5	70	47.5	10	75	2	"	44				
" 18	51	66	67.5	70	47.5	10	75	2	"	40				
" 20	52	66	67.5	70	47.5	10	75	2	"	43	15			
" 22	53	66	67.5	70	47.5	10	75	2	"	45	17			
" 23	54	66	67.5	70	47.5	10	75	2	"	46	13½			
" 25	55	66	67.5	70	47.5	10	75	2	"	44	14			
" 26	56	66	67.5	70	47.5	10	75	2	"	35	11½			
" 27	57	66	67.5	70	47.5	10	75	2	"	36	11½			
" 30	58	"	"	"	"	"	"	"	"	38				
7-2	59	"	"	"	"	"	"	"	"	41				
" 6	60	"	"	"	"	"	"	"	"	44				
" 7	61	"	"	"	"	"	"	"	"	41				
" 9	62	"	"	"	"	"	"	"	"	45				
" 11	63	"	"	"	"	"	"	"	"	40				
" 14	64	"	"	"	"	"	"	"	"	38				
" 16	65	"	"	"	"	"	"	"	"	41.5				
" 19	66	"	"	"	"	"	"	"	"	40				
" 21	67	"	"	"	"	"	"	"	"	37				
" 22	68	"	"	"	"	"	"	"	"	53				
" 24	69	"	"	"	"	"	"	"	"	40				
" 26	70	"	"	"	"	"	"	"	"	53				
" 29	71	"	"	"	"	"	"	"	"	29				
" 30	72	"	"	"	"	"	"	"	"	41				

Date 1915	Batch #	66° H_2SO_4	Acet Anilid	Nitrating a. 66°B H_2SO_4	Nitrating a. 70% HNO_3	Acetic Acid	Iron filling	Na_2CO_3	NaOH 20%	Para yield
8-2	73	66	67.5	70	48	10	75	2	125	41
" 4	74	"	"	"	"	"	"	"	"	46
" 8	75	"	"	"	"	"	"	"	"	55
" 10	76	"	"	"	"	"	"	"	"	92
" 12	77	"	"	"	"	"	"	"	"	37
" 14	78	"	"	"	"	"	"	"	"	40
" 17	79	"	"	"	"	"	"	"	"	42
" 20	80	"	"	"	"	"	"	"	"	44
" 22	81	"	"	"	"	"	"	"	"	40
" 25	82	"	"	"	"	"	"	"	"	40
" 27	83	"	"	"	"	"	"	"	"	46
" 28	84	"	"	"	"	"	"	"	"	47
" 31	85	"	"	"	"	"	"	"	"	60
9-17	86	"	"	"	"	"	"	"	"	40
" 21	87	"	"	"	"	"	"	"	"	40
" 24	88	"	"	"	"	"	"	"	"	40
" 26	89	"	"	"	"	"	"	"	"	43
" 28	90	"	"	"	"	"	"	"	"	35
" 29	91	"	"	"	"	"	"	"	"	43
" 30	92	"	"	"	"	"	"	"	"	40
10 2	93	"	"	"	"	"	"	"	"	42
" 5	94	"	"	"	"	"	"	"	"	43
" 7	95	"	"	"	"	"	"	"	"	41

Date 1915	Batch #	66° H_2SO_4	Acet Anilid	Nitrating 66°B H_2SO_4	Nitrating 70% HNO_3	Acetic Acid	Iron filings	Na_2CO_3 20%	NaOH 20%	Para
10-9	96	66	68	70	48	10	75	2	125	50
" 11	97	"	"	"	"	"	"	"	"	39
" 14	98	"	"	"	"	"	"	"	"	37
" 16	99	"	"	"	"	"	"	"	"	42
" 18	100	"	"	"	"	"	"	"	"	36
" 20	101	"	"	"	"	"	"	"	"	38
" 23	102	"	"	"	"	"	"	"	"	42
" 25	103	"	up to this	Batch	average	"	40 lbs per B			41
" 27	104	"	"	"	"	"	"	"	"	30
" 29	5	"	"	"	"	"	"	"	"	36
" 31	6	"	"	"	"	"	"	"	"	40
11-1	7	"	"	"	"	"	"	"	"	41
" 3	8	"	"	"	"	"	"	"	"	40
" 6	9	"	"	"	"	"	"	"	"	42
" 8	110	"	"							38
" 10	111									38
" 12	112									39
" 14	113									40
" 17	114									38
" 19	115									42
" 21	116									40
" 23	117									41
" 25	118									40

Date	Bale	H_2SO_4	Crude	Sm	x acid	Acet	Iron	$MgCl_2$	NaOH	Para yel	Bale	yel	Bale	yel	Bale	yel
11-27	119									40	143	40	167	42	191	35
	120									42	144	40	168	16	92	34
	121									42	145	43	69	13	93	36
	122									40	146	40	70	17	94	39
	23									40	47	44	71	14	95	39
	24									41	48	40	72	40	96	41
	25									40	49	43	73	43	97	38
	26									40	50	38	74	41	98	38
	27									40	51	40	75	40	99	
	28									41	52	41	76	36	200	
	29									42	53	42	77	32		
	30									40	54	40	78	40		
	31									52	55	38	79	38		
	32									48	56	40	80	37		
	33									42	57	41	81	37		
	34									36	58	40	82	46		
	35									38	59	40	83	44		
	36									40	60	39	84	38		
	37									40	61	39	85	38		
	38									41	62	39	86	38		
	39									38	63	17	87	41		
	40									44	64	46	88	30		
	41									42	65	12	89	44		
	42									44	66	40	90	39		

Paraphenylendiamine

#1 Batch

70 g p. Nitroaniline
40 " Iron
300 " HCl 40% conc
500 cc. Water

Started boiled 6 hr but Reduction not complete

Para-

#2 Batch

70 g p Nitroaniline
40 " Iron
20 " 25% Acetic Acid
500 cc. Water to 1000 cc.

boiled 6 hr. no all Reduced

Para

#3 Batch
70 g p Nitroanilin
60 " Zinc dust
2000 cc Water
make acid to Congo
(started but not finished) ex lost
boile 6 hr.

#4 Batch
same as #3 only using Reduced Iron
(started but not finished) ex lost
boiled 6 hr

Para

#5 Batch
70 g p Nitroanilin
400 g Fe_2SO_4 in 1-3 p of water
225 " $Ca(OH)_2$
400 cc Water

#6 Batch
70 gr p Nitroanilin
400 " Fe_2SO_4 in 1-3 p of Water
400 " Ammonia

Para

Batch #7

100 g Paranitroacetanilid
500 cc Xylol
250 g Iron filings
200 g 50% Caustic Soda
heat to 130°C with reflux cooler.

#1 - B7 In Glass flask
put in Iron & soda in 4 parts and allowed to boil between each charge all of the Iron and soda was added in 1 hours time with 15 min between each charge and then boiled 8 hrs
the reaction was not finished
stirring is necessary for this reac.

#2 (B7)
In Autopot with stirrer
an reflux condenser
200 g paranitroacetanilid
1000 cc Xylol
500 g Iron filing (all in at one time)
400 cc 50% Caustic soda added very slow
it took two hours to add it

Para

#2 - Batch
After soda was all in I gave it 4 hours at 115°C but then I found it was ready (now I allowed to filter air as it pleased leaving Xylol to go off through Condenser - and when 130°C attained I stopped and left stand over night
took out mush blew rest of Xylol over by steam and filter hot
boil the Crystals

sample in Bottle

Para

#3 same proportions as #2 and same conditions only stopped after 3 hours (temp only 115°C - and leaving at condition, left stand over night put at [illegible] bottom pot and add water and boil of glycol in air and filter hot. boil down to crystals sample in Bottle

Para

#4

1000g Paranitrotoluenesulf
250g Iron
1000 cc Water
200 g 50% Caustic Soda was slowly

This Batch was run with reflux cooler and temp was 100°C but gradually it went up to 105°C and at the end temp went up to 140°C when it was stopped
It was heated 20 hrs (Ammonia reaction)

boiled with water and steam drawn off
boil down - crystalize first crop - 36 g Dry
41 g wet
Mother liquor almost neutralized with HCl

no good

Para

#5

200 g P. Nitroacetanilid
500 " Iron
400 " 50% NaOH
1000 cc. Water

Started at 12.00 A.M. put gass on and 1/4 of the soda, but Motor stoped occasionally so I put in rest of soda in 3 different parts and by 2.00 o'clock it was all in and by 3.00 o'clock I had Tempt 130°C but lots of trouble with Motor, so stirring was not very effective at 7.30 pot started leaking and temp went down to 115°C - stood so till 9.00 o'clock when stoped let stay overnight - then boiled ~~down~~ with 3 liters of water let settle and siphon off - repeated twice more, then boiled all this liquor down adding 400 g of 32% HCl to the bulk. Crystalized Mother liquor 1100 cc. an Crystal 206 g wet and 158 g Dry - and after distilled in Vaccum. The Vaccum distillation was abolisyed

not reduced

Para

#6

200 g Paranitroacetanilid
500 g Iron
200 g 50% NaOH
1000 cc. water

} all put in at once

in this case I add 100 g 50% of NaOH to 1000 cc of water

started to heat & stir at 2.00 P.M. (shot off one flame gass) after 15 min. the tempt being 93°C - at 2.30 105 - here stirring was interrupted pot leaked - and Motor had to be fixed, at 3.00 stirred again tempt was 95°C. Delayed again 1½ hr. for same reason as first started again 4.30

not Reduced

Para

#7

same proportions as #6

but was run for 36 hrs when it showed no more yellowedness on which paper

Para

#8

same proportions except 400g of 70% Moth instead of 200g in #6-7 Batch

(started but not carried out)

[ITEM(S) FOUND IN BOOK]

68-

20 24 41

$C_8H_{10}NO + NaOH = C_6H_5(NH_2)_2 + NaC_2H_3O_2$

23
24
3
32
82
41

40-41

#1 p. Chlorinitrobenzol

100 g Benzol
216 g Nitrating Acid (10-15°)

1 h to run in Acid and run 2 h more
Tempt was 50° F when started but run up to 80° in 10 min and keep between 80-85°
Left all acid went in and then went to 100° Fh. and then down again
when ready to take off it was partly crystallized tempt was 83° Fh.

Washed water 3 times. Sod once - and again
" " 3 " each time 300 cc Water

Distilling starts to come over at 80°C
keeps at 95°C for some time then goes clear up to 140 - I stopped at 155°C
first fraction 13.4 gr

Freezing - to 15°C in Ice & Salt
Solid 46.5 g - M.P. 77-81 - Distill Vacuum 80-83
oil 42.7 gr

Solution of Acid 110 c.c.
100 ÷ 13 = 86.6 - 86.6 should give 121+
gave 89.5
loss 32

due to broke was uncovered - and then stirring

#2 p. Chlornitrobenzol

same portions as #1 - also Nitrating time but tempt was - 80-85 - while Nitrating and only went to 85° after which #1 - went up to 100° after Nitration
When finished all was oil and no Crystals at 75°Fh.

Washed 2 times Water each time 300 cc.
Neutralized Soda
Washed 1 time water

Solution of Waste Acid 113 oz. 96% 162.5
" " oil 105 " " 129.5

Distilling starts at 85°C and I stopped it at 180°C
Head 34.3 gr
solid 45.5 gr 78-83
oil 40.7 gram

100 ÷ 34 = 66 - 66 should give 92 gr
gave 86
loss 6 g

#3 o Chloronitrobenzol

Same portions as #1 and Nitrating time. On both #1 & 2 I used Ice to keep cool but on #3 only water is used (Tmp. 72°F) and Tmp. was kept at 85-111°F.
Fire showed a little better and it was run 1 hour longer after Acid was run in partly crystallized when taken out.
Tmp. was 85°Fm.

Washed 2 X Water
1 X Soda 3 c.c. 15%
" 1 Water

Acid 113 c.c. sp Gr 1615
product 103 "

Distillation - Crude 24.2 gr
Chile to 15° solid 53.4 gr M.P. 78-83
oil 47.4
some left in (Acid wast) ?

100 ÷ 24.2 = 75.8 - 75.8 shall give 106
it gave 100.8
5.2

#4 o. Chloronitrobenzol

Same portions as #1 - but nitrating was 9 hrs. an 3 hrs after and Tmp. was between 100-110 °Fm.
The whole contents was poured into 800 c.c. of Water & Ice Tmp. above 70°Fm.
washed 1 X 800 Water
" 1 " Soda 4 c.c. 15% NaOH
" 1 " Water
Then distilled same as #3

The total volume of Mixy Acid & Para is 220 c.c.

Heads 19.6 gr
Solid 54.3 " 78-83
oil 57.1

100 ÷ 19.6 = 80.4 80.4 shd give 112
it gave 107
5
loss

O. Chloronitrobenzol

#5

500 g Monochlorobenzol (using Hud #1-2-4 = 66.1 g
1080 g Nitrating acid (10-15)
acid run in, in 2 hrs, keeping tept, at 100-110° F then kept stirring 4 hrs after all acid was in.
The whole mass was put into water and was same as #4 only using 4 lts of water each time

Distillation was stopt at 150°C

Head 59.8 g
oil 317.4 g
solid 315 g m.P. 74-77

500 ÷ 59.8 = 440.2 440.2 solid gives 616
6.32
gain of 16

O. Chloronitrobenzol

#6

500 g same as #5 and using Hud #5 which is = 58.8 g
Acid run in in 2½ hr. tept went up to 140°F. once and to 130° once but ordinarily it was kept pretty low that is 85-100°F
Was run 4 hrs after all acid was in

after Distillation

Head 69.3 g
oil 259.5 g
solid 300 g m.P. 76-79

500 ÷ 69.3 = 430.7 420.7 solid gives 603
559.5
loss 43.5

For Fractional Distillation see over

Part [illegible] Tobacco

[illegible] Batch # 1[illegible] was [illegible]
[illegible] by [illegible]
on 27½" Vac Distilled at 155-160
[illegible] of 726.5g
[illegible] 4.5
730.5

Total [illegible] 732.2
[illegible] was [illegible] separate

Distillation of the oil (after [illegible]

1 fraction up to 200°C from [illegible] 711g
263.5g total distilled
52.3 solid liquor 207.3g

2 [illegible] 300g [illegible] 52.7g 247.3

3 " 300g solid 5g " 295g

4 " (255°C) 76.5g solid 49g

Fraction [illegible]
and after [illegible] distilled again [illegible] 45g
solid and [illegible] oil

155 to 160° - 27½" Vac

732.3

Heads 60.5
666.
726.5
tales 4.9
731.4

0.8 Loss

27½" Vac
Distilled at 155 to 160

732.2g crude

Heads 60.5
" 666.0
726.5
tales 4.9
731.4
732.2
0.8 Loss

Nitrobenzene $C_6H_5NO_2$ (123) Mw

Form.

78 g pure Benzol 90 g 70% Nitric Acid
100 g 66°Bé Sulphuric Acid

1 Batch
390 Benzol
450 Nitric
500 Sulphuric –

The acid mix when cold put onto Benzol also kept cold then left stand for several days then washed with water, first and then added a little soda to neutralize washed till neutral, then distill the Nitro benzol –

sp. g. 1st fraction 886 } 293
" 2nd " 914
" 3 1200 – 486 g

should give if 100% 615

therefor the yield now is 79%

$$C_6H_6 + HNO_3 = C_6H_5NO_2 + H_2O$$
78 63 123 18

Nitrobenzen

Batch # 2
1560 Benzol pure
1800 Nitric acid 70%
2000 Sulphuric " 66°Be.

I took 3 hours to stir in the acid and keeping kept no high the 25°C at one instant it went up to 41°C for a short while, then put on hot plate and rise the kept up to 80°C – which took 40 minutes and kept at 80°C – for one hour and then left stand on hot plate over night @ 60°C. – Nitric fumes was given, of all the time after washed and distilled it

yield 1435 g of Nitrobenzole
sp. g. 1200 – 1205 – between
205 – 220°C.

yield 58.3%

I noticed the wash water was quite yellow

Or must have made a mistake in weighing

Nitrobenzen

From (Book on Explosives)
As a rule 40 pts of HNO_3 1400 sp. g = 65.3 %
and 60 pts of H_2SO_4

or 100 pts of HNO_3 to 140 pts of H_2SO_4

an as a rule 3 pts of Mixed acid to
each part of Benzol

Nitration

When nitrating the tept must not
go higher than 25°C aporation
takes 8-10 hours then tept is
raised to 122°F and sometimes it go
to 212°F

if pure Benzol is used it
yields 152 pts of $C_6H_5NO_2$ for each 100 pts
of C_6H_6 instead of 157 theoritical

or yeild 96.4%

Nitrobenzen

Batch #3-

1560	Benzol	100%
1800	Nitric	70%
2000	Sulphuric	66°Be

Started to run in acid at 8.15 A.M. } 5 hrs
finished 1.15 P.M.
kept tept down to 20-25°C
than when all acid was in we allowed
Tept to raise up to 80-85°C and at
that time we put some water outside
the jar and then she gradually went
down, at 3.45 P.M. it was 59°C -
stirring all the time
at 5.00 P.M. tept was 42°C -, stopped
stirring, and kept stand till Monday
morning from Saturday 5.00 P.M.

after distilled
yeild 80% = 2000 gram

Nitrobenzen

Con 95

Batch # 1.

7.8 Lbs of Benzol (from Silver Lake)
9.0 " " 70% Nitric Acid
14.0 " " 66° Be Sulphuric Acid
Total Lbs of Acid 23 Lbs
all used but 1.6 Lbs. = .62 Lb HNO

Started at 9.45 A.M.
and finished at 6.P.M.
keeping kept between 10°-20°C and at last kept it go to 40°C - took it out into Jal and left it settle over night got 3 layers Nitro Benzol Acid of Sp.G. 1.625 = 76.74% H_2SO_4. and a layer of a whitish precipitate
4800 g Nitrobenzol
400 g Benzol not nitrated
and 1.6 Lb of Mix acid left
yield 98%

Anilin

Batch # 1 - from first Batch of my Nitro Benzol =

485 g of Nitrobenzol
40 lbs " HCl
40 " Water
520 g " Iron borings

Mixture put into Flask and put on hot plate and left there 50 hrs at that time the small $C_6H_5NO_2$ had gone then neutralized with sodaash and anilin blown out by steam

when separated from water yield 246 c.c. of Anilin sp.G. 1.00

after distilling a lighter liquid was obtained of same sp.G. as before and smelled Nitrobenzol,

$$C_6H_5NO_2 + 3Fe + 6HCl = C_6H_5NH_2 + 3FeCl_3 + 2H_2O$$

93

Aniline 2-23-15

Batch # 2

1200 g Nitrobenzen
480 cc. water
300 g Iron
120 g HCl
900 g Iron

Started 8.50 a.m. with 300 g Iron 2 H_2O started a little steam - Violent reaction, too much Iron - after reaction seemed finished 300 more of Iron but too much again smaller portion was added as follows

1100 a.m.	100 g Fe	Reaction complete at 5.00 p.m.
1145 "	100 "	
100 p.m.	100 "	
135 "	100 "	
230 "	100 "	
245 "	100 "	

1000 —

Aniline 3-19-15

Batch # 3

4800 g Nitrobenzol (4400 from #1 Batch in Big Pot
400 " from first Batches)
1900 cc. water
1200 g Iron } first charge
480 cc HCl
3600 g Iron added in small portions

Started at 9.15 a.m. to run in Nitrobenzol and Iron as follows

300 g Iron	at	9.30	} Iron added a little steam but too much as reaction got too violent - Temp = 95°C
300 "	"	9.45	
300 "	"	10.00	
100 "	"	10.35	
100 "	"	10.55	
100 "	"	11.00	
100		11.15	reaction kept about 98°C
100 "	"	11.27	
100 "	"	11.34	at 2 p.m. all Nitrobenzol in
200 "	"	11.40	[illegible]
200 "	"	11.45	amount [illegible]
100 "	"	12.15	Steam was at [illegible]
200 "	"	12.45	blow steam in but was
200 "	"	12.50	changed as pipe was
200 "	"	12.55	clogged up another was put
200 "	"	1.40	in an started again, but
200 "	"	1.48	
200 "	"	2.10	several times too much
200 "	"	2.20	steam carried over some
200 "	"	2.40	

of the slime - left all settle over night

for yield see page 94

18 L of water was used to blow over Aniline

Anilin

3-20-15
con
93

Batch #4

5000 g Nitrobenzol
1900 cc. Water
1200 g. Iron
500 cc HCl 20°Be = 32%
} first charge

Started at 1010 A.M.

300 @	10.35 @	40°C	when at 65°C Reaction started on vapors started and kept raised to 100 at 11.40 had to stop stirring
300	10.50	44 "	
300	10.55	48 "	
200	12.30	75 "	speed of stirrer Reduced to 125 RPM
200	12.45	87 "	time kept raised to 100° and
200	2.00	65	kept time up till 2.00 o'clock
300	2.15	54	so I put on cold water
300	2.25	58	which reduced kept to 54
300	2.35	67	(this was too low)
200	2.50	75	at 4.30 P.M., started to blow
200	3.15	93	over the Anilin but distillate
200	3.50	99	was not clear so returned
200	4.00	98	it with 400 g of Iron and
300	4.50	100	stirred again till 6.00 o'clock
100	5.10	100	when I started to blow over

the Anilin which lasted 3 hours - kept 100°C
18 liters of water was used to blow over the anilin

for yield see page 94

Acetanilid

279 gram of Anilin (Eimer & Amend)
600 " " 99% Glacial Acetic Acid
boiled 24 hours
poured into water which precipitates it washed free from acetic and dried
yield 325. about 10% left in Mothliquid

Anilin 3-22-15

Batch # 5.

Proportion of first charge same as #4 Batch started at 1000 A.M. - heated the pot up first then put in first charge and as follows.

10.20 A.M.	400 g	Drm		at 97°C Reaction begins
1040 "	400 "	"	68°C	here it was allowed to its
1110 "	200 "	"	97 "	own reaction till 700 gms
1115 "	200 "	"	96	the pot was then 52-0°C.
1130 "	200 "	"	94	with steam was blown in to
1145 "	200	"	94	test for clear anilin but still
1200 "	200	"	94	showed a lot of Nitrobenzol
1215 "	200	"	96	& then added more drm an
1230	200	"	96	
1245	200	"	96	
100	200	"	96½	1000 HCl an stirred again
115	200	"	96	till S.G. 0.9827
130	200	"	96	then it showed good
145	100	"	96	clear anilin color
200	100	"	96	
215	100	"	96	and then started to blow
230	100	"	96	
245	200	"	90	The anilin oil - pot was
300	200	"	91	more full than the "
330	200	"	95	usual
720	200	"	52	

Trouble by blowing over Anilin - after first 4-5 liters of Anilin + Water had come over - she came over black - an some blew out of Corkshot so I had to draw off 6 L of water from pot and then run again - till black started again then left stand over night an then drew of 2 gal of water again started to blow over last of the Anilin 4 gal of water off the pan an 1 gal washed at last (yield Pg. 94)

Anilin

yields of Batch # 3-4-5

#3 Crud anilin 2835 gram 95% Benzol
" 4 " " 2,900 " " "
" 5 " " 2325 " Pure "

Distillation

						Anilin
#3	@ 98°-130 -	Water anilin	8½ %	@ 183°C	90%	1 3/7
" 4	@ " "	" Benzol	7½ %	@ 183	90%	2 1/29
" 5	@ " "	" "	7 %	183	90%	?

Yield of Pure Anilin

		Theoretical
#3 - 2835 g Crud anilin		= 78%
450 " Anilin in 18 L of Water		total 90.4%
#4 2900 g Crud anilin		76.7%
450 " Anilin in 18 Lits Water		88.7%
#5 2325 " Crud anilin		61.5%
978 Anilin in 33.6 L Water		87.5%

#5 Batch I believe was a good reduction and would give full yield - had not the numerous difficulties arisen from too little space for steam

Nitrobenzen

Batch # 2 -

proportions a duplicate of # 1 ?
Acid was run in and kept was
kept between 20-25°C and at last
was allowed to raise to 45°C
Mean run 1 hours after - the whole
operation took 5 hr,
except using 15½ Lbs of H_2SO_4
and Batch # 1-2 - was made of
90% Benzol
This Batch ~~A~~ looks good
Acid had a sp Gr of 1.610 and
Nitrobenzol had a sp Gr. of 1.185
before washed - washed twice with
water and then soda added and washed
several times

yield 5-200 grams = 94.5%

Nitrobenzen

Batch #3 -

18 Lbs of 66° H_2SO_4
9 " 70% HNO_3
7.8 " pure Benzol
having same condition and kept as
2 Batch

Sp Gr of Acid left = 1.660

Yield 5100 gram = 94%

Nitrobenzen

Batch #4

10 Lbs of Benzol pure
11.5 " " Nitric acid 70%
20 " Sulphuric " 66°Be

run same as other Batches
but kept only warm up to 37°C

Acid has a sp.g. of 1.635

Distilled yields 6330 g Nitrobenzol
85 g Benzol
20 c.c. Residue

yield 90.1% from 6330 g

Nitrobenzol

Formulas

$C_6H_6 + HNO_3 + H_2SO_4 = C_6H_5NO_2 + H_2O + H_2SO_4$

10 Lb (11.5-70%) (20-95%) 15.07 2.3 25.7-74%
(8.1 HNO_3 + 3.4 H_2O) (19 H_2SO_4 + 1 H_2O)

1000 1150 2000 1507 230 2570

$NaNO_3 + H_2SO_4 = HNO_3 + NaHSO_4$
85 98 = 63 120
10.9 13.-95% = 8.05 15.4 (= 10 Lb Benzol)

1090 1300 8.05 1540 1000 Benzol
↳ 20 lbs add 2000 95% H_2SO_4

[illegible] Nitric 70%
what can you [illegible]
what cost $NaNO_3$
and what would it cost to make nitric
Can we use the acid for carbolic

Total 500
flask 46.8 g

4-5-15

Nitrobenzol from $NaNO_3$

Nitrating acid made by Mr. King

Batch #6 total acidity 91% of HNO_3 and 66% H_2SO_4
" " 7 " " 85 " " 53 " "

Batch #6 contains	298 g HNO_3	+	892 gr H_2SO_4	Acid total	1,310 g
" 7 "	518 "	+	1052 "		1847
total	816 "		1944		3157

78 - 63 = 1,010 = 816 (Benzol)
10 - 20 = 1010 = 2020 say OK

Could not get tipt up - (only 30°C)
waste acid is 1550 sp gr
amm Nitrobenzol 1150

by distillation I got 465 cc Benzol
" " " 640 g Nitrobenzol

4-6-15

Nitrobenzol

Batch #5

20 lbs of Sulphuric Acid 66°Be } = 2.36 gal
115 " " Nitric " 70% }
10 " Benzol pure = 1.42 gal
total 415 lbs

Started at 1.40 P.M. - at 430 cooling water was stopped and 1½ L of acid was left to run in yet, at 442 all acid was in and tipt was 47°C and raised to 57°C at 455 - was stirred till 600 when allowed to settle till 800 P.M. The tipt, while adding acid run from 20°C to 43°

Pot capacity 4¾ gal to brim
Cooling surface 561 sq in outside
20 gal of water to cool to keep 25°C
the water was 67°Fh. when running into sink
Total acid & Nitrobenzol when finished 3.7 gal
and space left in pot was 2½ in to brim
or with pot = 4.3 gal and 3.78 gal used = 1 gal space left
Stirred 3 hours to add the acid (2.36 gal)
except the last ten min water was stopped
and the last 1½ liter was run in which helps
to rise tipt (no fume was coming out
in refluxcooler - it did not even get warm.

Paraphenylendiamine

Nitrating Pot 250 gal. cap. = 1000 Lbs = 75 gal
255 Liters H_2SO_4 66° Be
246 Lbs of Acetanilid
462 Lbs of Nitrating Acid

Compared to our present 65 gal. cap.

1

Theoritical proportion
165.5 Liters of H_2SO_4 66° Be = 44 gal
170 Lbs of Acetanilid
79.5 Lbs of Nitric acid

(23 Lbs of water formed)

2

25 Lbs of Acetic Acid 28%
190 Lbs of Iron filings
10 Lbs of Soda Ash

3

325 Lbs of Caustic Soda 20%

Should give 100 Lbs Para

and as a biproduct 125 Lbs of Acetate of Soda

(1000 Lbs of Sol) Filter bags for acetyl & for cailefying

Acetanilid

Copy from Fred Otto & his pot has 15 gal cap.

55 Lbs Oil
60 Lbs Acetic Glacial

Yield 70 Lbs 14 oz of Crud Acetanilid
he put in 12 Bottles of acid glacial
" got out 7 " " (acid Recovered %?)

Starter	Tempt °C	Tempt °C	
4.45 P.M.	40	40	1 hr – 40°–100°C
6.00 "	100	100	1 hr 100–120°C
7.00 "	120	120	3 hr 120–120°C
8.00 "	120	122	1 hr 120–150°C
9.00 "	120	122	1 " 150–175
10.00 "	120	126	1 " 175–200
11.00 "	150	150	1 " 200–250
12.00 "	175	175	1 " at 250
10 P.M.	250	220	—
10.00 "	250	250	
2.00 " (Cool 1 hr)	250	250	

Recovering of Acetic Acid
as Chas Dally Think
Test Run

2 Lbs of oil
2 Lbs of Acid glacial acetic } yield 1137 g
and 625 g of Oil & Acid

To recover acid & oil
add 350 c.c. of 50% NaOH to the acid and oil or till you turn Red litmus blue Draw off oil and evaporate till Dryness then fuse - to 500 g of fused ~~salt~~ add 750 c.c. H_2SO_4 66° Be and Distill the acetic acid

Acetanilid
To make 200 Lbs per day

157 Lbs of Anilin oil
164 Lbs of Glacial acetic acid

240 Lbs of oil & Acid to be recovered
Neutralize with 57.6 Liter of 50% Lye

to my 500 g fused salt add 750 cc H_2SO_4

if above figures 240 Lbs + 57.6 L = 300 Lbs

then we will require 225 Liters of H_2SO_4 66° Be

800 gal of Benzol = 6000 lbs

7 × 500 gal pots for Nitrating
each pot for 1160 lbs of Benzol
and — 3000 " Nitratig Acid
= 130 gal Benzol
= 220 gal Nitratig acid

3 settling tanks each 700 gal
for washing

1. 1200 gal Still Boiler plat
Still gives 9000 lbs Nitrobenzol

Aniline

7 Reducing stills 1000 gal each
each equipped with steam condenser piping
settling tanks

1 Vacuum Oil pump 5-10 mm
1 Prim Pump 3 atms

Lbs 6000 Benzol + 18000 lbs Acid
9000 Nitro benzol + 138 lb of Water + H_2SO_4 8%
1160 lb Iron 1000 lbs HCl
15 men

Nitrobenzene

Items	#1 90%	#2 90%	#3 pure	#4	#15
Benzol pure	7.8 Lb	7.8 Lb	7.8 Lb	10 Lb	10 Lbs
Nitric 70%	9 Lb	9 "	9 "	11.5 "	11.5 "
Sulphuric 66%	14 Lb	15 "	15½ "	20.0 "	20.0 "
Nitrating Tempt	10-20	20-25	25	25	20-35 (45)
End "	40° C	45°	47° C	37° C	57
Cap. of pot	4 3/4 gal	=	=	=	=
Benzol in space					1.42 gal
Acid space					2.32 gal
spare space					2½ "
Benzol wt	400 gr	?	?	?	
Acid wt	1.6 Lb	?	?	?	none
Lbs Waste acid					26
" sp. gr.	1625	1650	1660	1635	1610
" %	70.74				
Nitrobenzene	4,800 g	5,200 g	5700	Distilled 6330	
yield	98%	95%	94%	90%	
Nitrating time	5 Hr	5 Hr	6	7	3 Hr
Water used per Hour					20 gal
" Tempt					67° F
sp in of pot					561 gal

Aniline

Items	#3	4	5	6	7	8
Nitrobenzol	4,500 g	4,500 g	5,000 g			
Iron	4,500 g	4,500 "	5,400			
Water	1,500 cc	1,500 cc	1,700 cc			
HCl %	450 "	450 "	470 "			
Lime						
Temp °C	100 °C	100 °C	95 °C			
Time Hrs	5½	7	10			
Reduction Space	2 gal	2¼ gal	water to much			
After anilin space	2¾ "	3½ "	"			
Lime space			1.3 gal			
Cap. of Pot	4¾ gal	=	=			
Ra. Reaction						
Temp	100 °C	=	=			
Time	3 hr.	occasional	3 hr.			
Raw Stuff						
Water for dilution	4½ gal					
Aniline	283 g	290 g	233 g			
Yield crude	90.4	87.7	87.5			

Aniline

Material	Where used	Price	Lbs used	Total $	Remarks
Acetanilid		.34	67.5	22.95	
Nitric Acid 42° Be		.05 1/4	46.5	2.44	
Sulphuric " 66° Be		.0083	360.0	2.99	
Acetic " 28° Be		.02 1/2	20.0	0.50	
Iron borings		.0045	75.0	0.34	
Soda ash 58%		.0069	7.0	0.05	
Caustic soda 20%		.017	200.0	3.40	

Acetanilid + Nitric acid = Paranitroacetanilid
675 lbs 315 - 90 - % ortho 20% ↓ 72 lb
C_8H_9NO HNO_3 $C_8H_8N_2O_3$

Acetic Acid + Iron borings = Ferrous Acetate + Hydrogen
20 lbs 38% 75 lbs
$CH_3(COOH)$ Fe $Fe(C_2H_3O_2)_2 + H_2$

+ [Paranitroacetanilid = Acetylparaphenylendiamin
lbs 90 - % of ortho lbs 65 or 75
$C_8H_8NO_3$ $C_8H_{10}N_2O$

Soda ash + Acetate-Acetic Acid = Soda Acetate
7 lbs
$Na_2CO_3 + CH_3(COOH) = NaC_2H_3O_2 + CO_2$

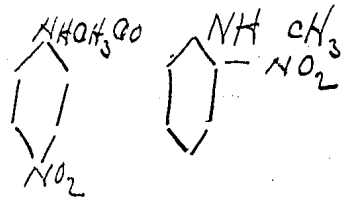
Acetylparaphenylendiamin + NaOH = Para
~~65~~ lbs 75 lbs 25 lbs 54
$C_8H_{10}N_2O$ NaOH $C_6H_4(NH_2)_2$

68 - + 20 = 54 + 34
$C_8H_{10}N_2O$ NaOH $C_6H_4(NH_2)$ + $NaC_2H_3O_2$

P.S. If 20% loss of in ortho yield = 43.

Nitrobenzol + C_2H_2Cl + NaOH
61.5 84 90

Measuring tanks piped and measured
Filter cloth for Centrifuge and for Reducer
200 Lbs of Acetic acid pr day Glacial
1000 Lbs of Ice
300 Lb. Caustic Soda 70% pr day
Thermometer for Nitrator –
See Hunter how to run oil burner
~~See~~ how to run up H_2SO_4
and what to do with water condensed
from Reducer – and box for brush)
About Drying pan & salt



26 Sp.Gr = 1700 · 1 liter

100 - 26 lbs

26) 7900 (304 lbs of acid
78 — 81 Litre of acid
100
104

304
454
1216
1520
1216
17) 138076 (81.
136
20
17
3.1

4/81 (20

85
01
129 —

Acid Cooled pots 125 gal
Measuring tank for Nitrating 41 gal
Nitrating pot 258 gal
Precipitating 950 gal
Reducer 600 gal
~~Saponifiers~~ 650 gal
Caustic tank 850 gal

10-

96 - 200
27 1
13

1

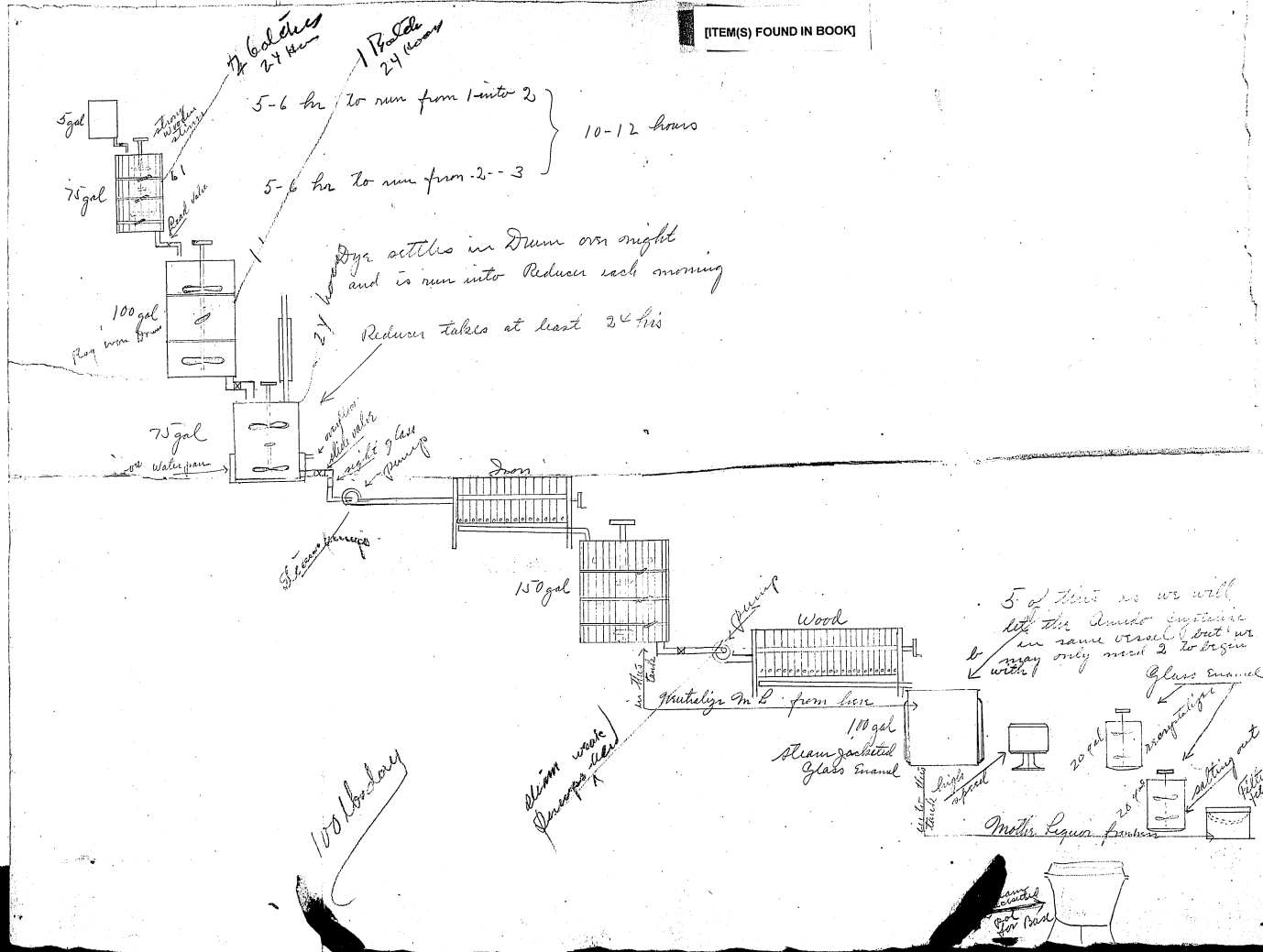
1

1 1

(15 3.26)

(856.06) for 1 week

[ITEM(S) FOUND IN BOOK]



[ITEM(S) FOUND IN BOOK]

Monocalcium Phosphate
Melts Easily —

Metaphosphate of Copper
insoluble in acids or
alkali
See Watts Dic. under
Phosphoric acid page
560 -

[ITEM(S) FOUND IN BOOK]

Pyrophosphate of Magnesium fairly fused

Neutral phosphate of Zinc — Trizinc Salt
Easily melts to transparent glass insoluble in water

Papers 5 & 7
Glaze on Crockery

1 part Clay 1 Feldspar 20g
2 Borax 20g

Powder off then 180 mesh
pack hard after slightly moistening
a fuse —

Then repowder & determine
Melting point —

[ITEM(S) FOUND IN BOOK]

12/16/15

Christensen

Look into this, I think there is a mistake somewhere in Opdykes accounts

Mr Meadowcroft It dont seem possible that it costs $2.85 per pound,

Edison

Just as a reminder

We have been billing the Disc Record Mfg Division for all Cera so far delivered to them at $2.50 per lb.

It Cost to make it in October $2.55 per lb. They understand our billing so far is subject to change

?

Opdyke